Morphology and Properties of Low Density Polyethylene Reinforced with Thaumatococcus Danielli Composites

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Abstract

The morphology and properties of a recycled Low Density Polyethylene (LDPE) reinforced with *Thaumatococcus danielli* (TD) petioles has been studied. The physical and mechanical properties measured include: water absorption, hardness, modulus, tensile strength and impact energy. The wear behaviour was characterised using linear regression and analysis of variance (ANOVA). The microstructure of the LDPE-TD composites produced was examined using scanning Electron Microscope (SEM). The results revealed that the addition of TD petioles increased the tensile strength, hardness and modulus with slightly reduction in impact energy. The improved strength and hardness was attributed to the presence of second phase TD petioles particles which fairly increased interfacial bonding at the particle-matrix interphase. The main effect of TD petioles additions and load variable had a pronounced effect on the wear behaviour of the LDPE-TD composite, while the interactions between the sliding distance and time had the most significant effect on the wear behaviour of the LDPE-TD composite. Hence, *Thaumatococcus danielli* (TD) petioles particles can be used in increasing the properties of polymer composites.

Keywords

Tribological; Thaumatococcus Danielli; Mechanical Properties and Wear

Introduction

Polymer matrix composites increasingly employed in industrial application because of their unique combination of mechanical, electrical and thermal properties, have high specific strength and modulus, excellent fracture toughness, fatigue properties, good corrosion resistance, thermal and electrical resistance properties(Tong et al, 1999, Wambua et al, 2003). This combination of properties, particularly their high strength to weight ratio, make them very attractive materials for transport applications where energy efficient materials become most relevant. Thermoplastics increasingly displace metals in many gears and bearing applications. The ability to absorb shock and vibration and to operate with less power and noise without lubrication are advantages that thermoplastics hold(Wang et al, 2001, Jin et al, 2003). Mechanical properties of composites are strongly dependent on the reinforcement and interfacial compatibility of the composite system.

Several natural fibers have been used as reinforcement in polyethylene due to low cost, density, resistance to breakage during processing, energy content and recyclability(Liming et al, 2007, White and Ansell, 1993, Hornsby et al, 1997, Mwaikambo and Ansell, 2002, Mishra et al, 2002). Field crop residues and/or agricultural by-products such as cereal straw, cornstalks, flax straw, corn cob, rice husk and bagasse represent a potentially valuable source of fiber which could be used as a supplement or a direct substitute for wood fiber in the manufacture of composites(Satyanarayana et al, 1999, Bledzki and Gassan, 1999, Hirao et al, 2003). Many authors have reviewed the latest developments in the application of natural fibers(Nor Azowa et al, 2010, Hinrichsen et al, 2000, Agunsoye et al, 2012). The widespread investigations on the preparation and properties of thermoset and thermoplastic composites with the application of natural fibers such as kenaf , jute(Mwaikambo and Ansell, 2002), sisal(Satyanarayana et al, 1990), straw(White and Ansell, 1993), coconut shell(Agunsoye et al, 2012a), palm kernel shell(Agunsoye et al, 2012b) have also been carried out. The natural fibers are used for variety of appliances such as packaging, low-cost housing and structures and the use of agricultural crop residues could boost rural agriculture based economy.

Environmental regulations and ethical concerns have triggered the search for materials that are environmentally friendly. As we push towards making the world 'green', the need for global participation in saving our environment and making the world more affordable to live in cannot be overemphasised. A pressing issue in developing countries today, is the recycling of waste products and other agricultural by-products suitable for the invention and characterization of new materials(Agunsoye et al, 2012a). Since they are waste, the utilization of natural fibres as reinforcement for polyester composite an available eco-reusing technique, Disadvantage of these fibres are that the consistency of the fibres cannot be guaranteed; along with the sensitivity to the moisture absorption from the environment, and that they do not adhere well to a polymer matrix under moist conditions (Agunsoye et al, 2012a).

Polymeric materials are increasingly used in a wide range of applications where resistance to wear is important. The application of the use of polymer composites has been extended to mating parts of machines where wear is a critical requirement and biomedical joint replacement as well as glazing materials where damage results in loss of optical properties(Manikandan et al, 2002, Monteiro et al, 2008, Bodros et al, 2007). Polymers are ideal materials for bearing applications due to their general resistance to galling and seizure, their tolerance to small misalignments and shock loading and their low coefficients of friction; as glazing materials, their low density and high toughness (compared to traditional glass) are desirable properties(Roberts and Wiche, 1993). In many applications, polymers may be subjected to abrasive wear, often due to contaminants within a system, and such abrasion may result in loss of function(Friedrich, 1986). While attention of academia and industry on material properties is largely focused on mechanics, *wear* causes losses in industry at least not smaller than fracture caused by mechanical deformation(Roberts and Wiche, 1993). Studies on polymer matrix composites subjected to sliding and abrasive wear indicate that wear resistance depends on the detailed properties of the material as well as the external wear conditions such as applied pressure and contact velocity (Roberts and Wiche, 1993). Researchers have shown that tribological behaviour of polymers may be improved by filling them with inorganic particulate compounds or fibers(Roberts and Wiche, 1993, Monteiro et al, 2008). The wear resistance of ultra-high molecular weight polyethylene has been found to be much higher than that of carbon steel and bronze in sliding friction, as well that of nylon-66, teflon, carbon steel, ceramic and enamel coating under such abrasive wear conditions as water–sand slurry or soil(Mohanty et al, 2000, Voss and Friedrich, 1985, Tanaka, 1986).

Thaumatococcus danielli (TD) species of tropical flowering plant are found in the rain forest of Ghana, Nigeria and surrounding African nations. Commonly called 'ewe moi-moi' by the Yoruba in western Nigeria, the study leaf petioles are used as building materials, and the leaves are used to wrap food. TD seeds have a number of traditional medicinal values and uses. This study seeks to investigate the mechanical and wear behaviour of recycled low density polyethylene composite reinforced with TD petioles (LDPE-TD). This is expected not only to overcome the pollution and waste management problem across our cities associated with improper disposal problem, but also to add value and extend the usefulness of TD waste as reinforcement in low density polyethylene (LDPE) for some tribological applications.

Methodology

The petioles of *thaumatoccocus danielli* were trimmed, oven dried in a Carbolite Furnace at 45°C for 10 minutes to remove the moisture grinded and sieved into 40μm powder (Figure 1). The particulate TD petioles and the LDPE were blended together using a two-roll rheomixer at 50°C and a rotor speed of 60 rpm. The percentage of the TD in the matrix was varied from 2% to 8% to produce four different compositions. Compression of the composites was carried out with a Wabash machine V200 hot pressing machine for 7 minutes under controlled pressure (30 tons) at 150°C. Before compression, the samples were insulated in an aluminium foil to prevent the composite from sticking to the die mould. Each of the samples was cooled to room temperature under sustained pressure before it was removed from the press. Before testing, all samples were conditioned for 72 hours at a temperature of 25°C ± 2°C and a relative humidity of 55% ± 5%(Mishra et al, 2002).

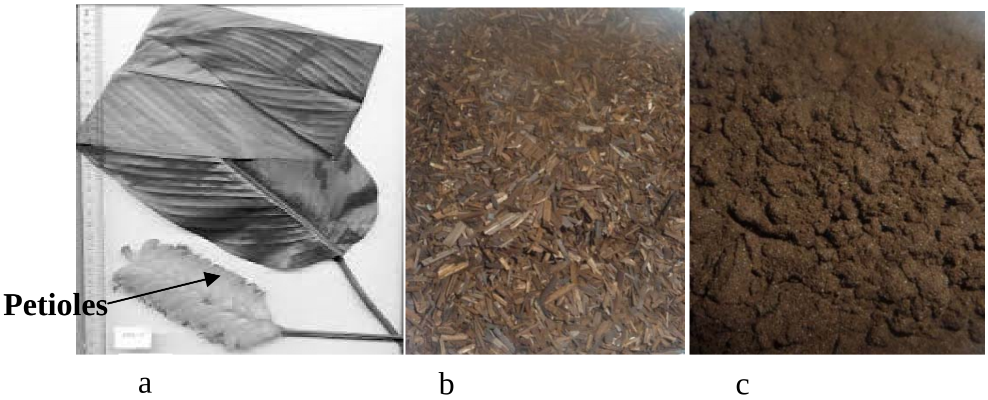

FIG.1 PHOTO OF THAUMATOCOCCUS DANIELLI: (A) STALK AND LEAVE (B) OVEN-DRIED PETIOLES (C) GRINDED PETIOLES

Scanning Electron Microscope (SEM), model EVO-MA10 LaB6 Analytical VP-SEM was used to determine the morphology of the composite samples at 20 Kv. The results of the SEM micrographs are presented in Figures 2-4. Necessary precaution was observed so as to increase surface conductivity.

The tensile test of the composite samples was conducted on an Instron testing machine with a strain rate of $2x10^{-3}s^{-1}$ as specified by the American Society for testing and Materials(Agunsoye et al, 2012a).

The impact test of the composite samples was conducted in accordance with ASTM D256-93(Agunsoye et al 2012b) using a fully instrumented Avery Denison test machine. Charpy impact test was conducted on notched samples.

Hardness of the composites was determined by Rockwell hardness machine (BS903 part B 26) [15] using 1.56 mm steel ball indenter, minor load of 10kg, major load of 100 kg. Before the test, the mating surface of the indenter, plunger rod and test samples were thoroughly cleaned.

In order to determine the rate of water absorption of the samples, initially weighed dried samples were placed in a beaker with water and reweighed at an interval of 24 hrs for 7 days (168 hrs). The water absorption rate was determined using Equation (1).

$$\%\ \text{weight gained} = \frac{\text{final weight} - \text{intial weight}}{\text{initial weight}} \quad (1)$$

A pin-on-disc apparatus was used to investigate the dry sliding wear behaviour of the fabricated polymer composite. A 150 μm mesh emery paper was stuck on 200 mm diameter plate pin-on-disc apparatus. The experiment was carried out with variying sliding distance, time and load. The weights of the samples were taken before and after the wear test with an electronic scale with accuracy 0.001 mg and the difference between the two weights was used to determine the wear volume loss. Subsequently, the wear rate was calculated using Equation (2). After each test, the worn-out debris was cleaned from the emery paper with a compressed dry air blower before subsequent test. In order to investigate the interactive effect of two or more process parameters on the wear behaviour, experiments were conducted in accordance with standard 1.8 orthogonal array, with a view to investigating which of the design parameters; sliding distance, load and time has the most significant effect on dry sliding wear(Tanaka, 1986).

$$\text{Wear Rate} = \frac{\text{wear volume loss}}{\text{sliding distance x applied load}} \quad (2)$$

Factorial design and linear regression methods are common tools used in engineering analysis for the data acquisition. Two levels of each of the four factors were used for the statistical analysis. The levels for the four factors were entered in Table 1. A model was used to represent the wear rate, W as a linear function of the process parameters in Equation 3: where (Sd) represents the sliding distance, (Tt) for time, (ld) for the load and the percentage composition of the reinforcer represented by (Rr).

$$W = f(Sd, Tt, ld, Rr) \quad (3)$$

The general model is represented as shown in Eqn. 4.

$$\begin{aligned} \mathrm{W} = \ & \beta o + \beta_1 \mathrm{Sd} + \beta_2 \mathrm{Tt} + \beta_3 \mathrm{ld} + \beta_4 \mathrm{Rr} + \beta_5 \mathrm{SdTt} \\ & + \beta_6 \mathrm{Sdld} + \beta_7 \mathrm{SdRr} + \beta_8 \mathrm{Ttld} \\ & + \beta_9 \mathrm{TtRr} + \beta_{10} \mathrm{ldRr} + \beta_{11} \mathrm{SdTtld} \\ & + \beta_{12} \mathrm{SdTtRr} + \beta_{13} \mathrm{SdldRr} \\ & + \beta_{14} \mathrm{SdldRr} + \beta_{15} \mathrm{SdTtldRr} \quad (4) \end{aligned}$$

Where βo is average response of W and $\beta_1, \beta_2, \beta_3, \beta_4, \beta_5, \beta_6, \beta_7, \beta_8, \beta_9, \beta_{10}, \beta_{11}, \beta_{12}, \beta_{13}, \beta_{14}, \beta_{15}$, are coefficients associated with each variable Sd,Tt, ld,Rr and interaction.

TABLE 1 PROCESS PARAMETER FOR THE WEAR TEST

Factor	Name	Unit	Low level (-)	High level (+)
Sd	Sliding distance	M	70	140
Tt	Time	Sec	60	120
Ld	Load	N	8.33	10.29
Rr	Reinforcer	wt%	0	8

Result and Discussion

The rate of water absorption by the composite over a period of 168 hours at an interval of 24 hours increased with rising filler percentage in the composite. The increased water absorption was due to the hydrophilic nature of the TD petioles (Figure 2). This is due to the absence of water repellent agents in the composite manufacturing. The agro-waste particles affected the water absorption properties negatively. The similar results were found by Agunsoye et al(2012a).

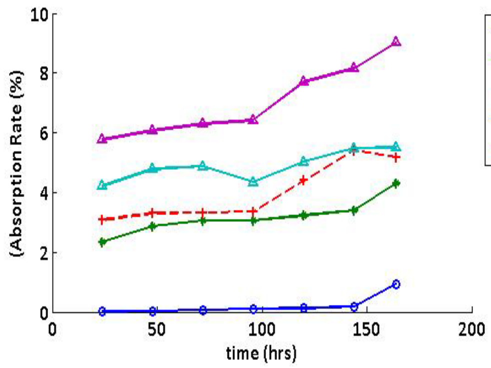

FIG. 2 ABSORPTION RATE OF TDPE-TD COMPOSITES

The swelling that occurs during the water absorption is the sum of two components, namely, swelling by hygroscopic particles and the release of compression stresses imparted to the composites during the pressing of samples in the hot press [10]. The results obtained is at par with the work of Satyanarayana et al.(1990), who also found out that the water absorption of the high–density polyethylene/egg shell composites increases by the rising exposure time for the same filler content.

The hardness of LDPE-TD composite increased with increment in the filler content of the composite. A maximum value of 6.67HB was obtained for the sample with 8% volume fraction of filler in the composite (Figure 3).

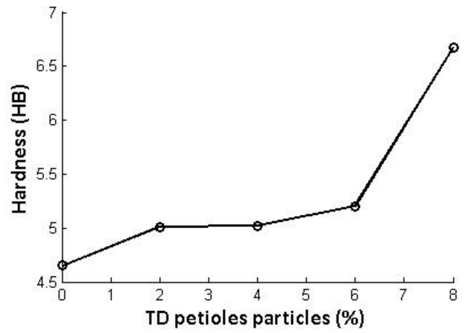

FIG. 3 VARIATION OF HARDNESS VALUES AGAINST TD PETIOLES PARTICLES

The hardness values of the composite samples rises as the percentage breadfruit seed hull particles addition increases in the polymer matrix (Figure 3). This is due to increment in the percentage of the hard and brittle phase of the ceramics body in the polymer matrix. In comparison with the unreinforced polymer matrix, a substantial improvement in hardness values was obtained in the reinforced polymer matrix. This is in line with the earlier works of Agunsoye et al(2012b), and Satyanarayana et al(1990).

The Young Modulus, which is a measure of the rigidity of the samples, is significantly affected by the volume fraction of TD petioles particles within the composite. As the filler content rises the rigidity of the composite increased (Figure 4). The increases in modulus elasticity with increasing TD petioles particles addition is expected since the addition of TD petioles particles to the polymer matrix increases the stiffness of the composites(see Figure 4). The presence of polar group in the polymer matrix may contribute to electrostatic adsorption between polymer and the particles. This phenomenon is driven by different charges acting on matrix or particles surfaces; which depends on particle type, pH value or inter-medium**(**White and Ansell, 1993, Nor Azowa et al, 2010**)**. This mechanism strengthens the composites interface, hold them together and increase their resistance to deformation **(**Agunsoye et al, 2012b, Manikandan et al, 2012 **).**

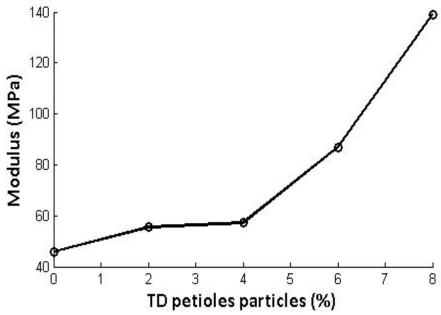

FIG. 4 VARIATION OF MODULUS AGAINST TD PETIOLES PARTICLES

The tensile strength of the composite consistently increased as the filler percentage increase in the composite. A maximum stress value of 0.32 MPa was obtained for the sample with 8% filler in LDPE-TD composite (Figure 5).

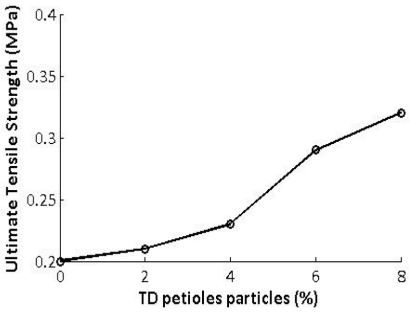

FIG. 5 VARIATION OF TENSILE STRENGTH AGAINST TD PETIOLES PARTICLES

As the TD petioles particles loading increased, thereby increasing the interfacial area, there was fairly good interfacial bonding between the hydrophilic particles and hydrophobic matrix polymer, which leads to increment in the tensile strength[15-19]. By applying the rule of mixture, the increased hardness can be attributed to the filler which has higher hardness value. The sample with the highest percentage of filler (8%) in the matrix has the lowest impact energy (Figure 6). The decrement in impact energy with increasing particles loading might also be due to the decreased deformability of a rigid interface between the particles and matrix.

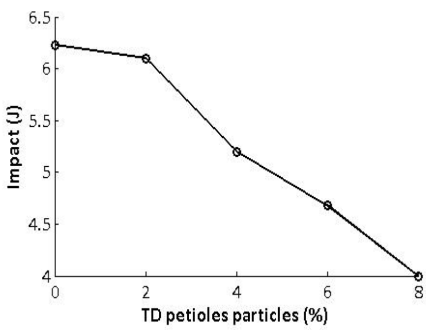

FIG. 6 VARIATION OF IMPACT ENERGY AGAINST TD PETIOLES PARTICLES

The polymer matrix is the sample without filler (TD petioles) additions. It is visible to see that the microstructure is homogeneous (Figure 7).

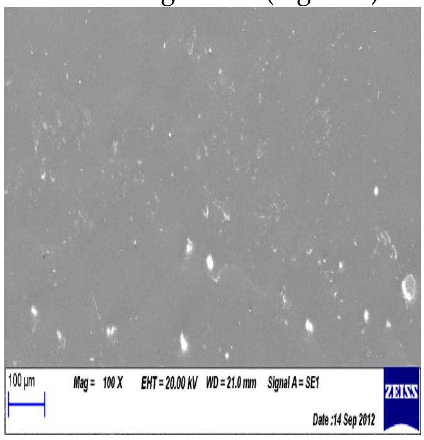

FIG. 7 SEM MICROGRAPH OF POLYETHYLENE SAMPLE WITHOUT FILLER ADDITIONS

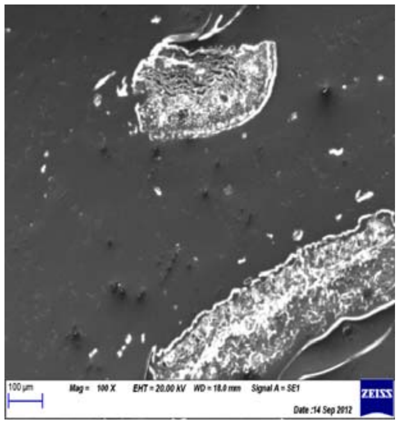

FIG. 8 SEM MICROGRAPH OF TDPE COMPOSITE WITH 2% OF TD PETIOLES PARTICLES

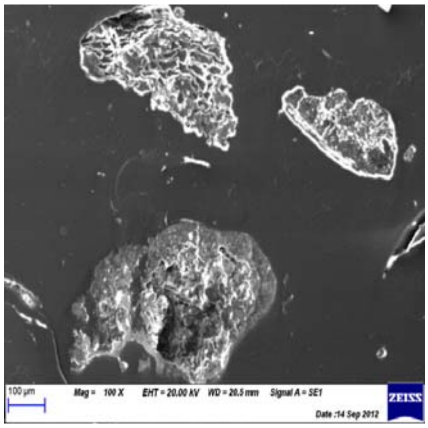

FIG. 9 SEM MICROGRAPH OF TDPE COMPOSITE WITH 8% TD PETIOLES PARTICLES

Figures 8-9, represent the SEM micrograph for the two extreme compositions of the developed composite. The micrographs indicate segregation of the TD petioles within the matrix of the composite which led to clustering of the particles. This phenomenon led to inhomogeneity of the composite. Despite the observed inhomogeneity, the composite attained a fairly higher

strength as the percentage of TD petioles filler increase. Hence, the properties of the composite can be optimized by the additions of anti-clustering agent. Also, the decreased observed in toughness can be explained by the morphology of the composite. By implication the strength of the composite can be sacrificed slightly for increased toughness by the additions of anti-clustering agent in application where strength is not a primary consideration. This would guarantee homogeneity of the blend and increase flowability during production of intricate shapes with fewer defects.

The analysis of variance was used to investigate which design parameters significantly affect the wear characteristic. It was accomplished by separating the total variability of the wear results, which was measured by sum of the squared deviations from the wear rate obtained, into contributions by each of the design parameters and the errors. Substituting the coded values of the variables for any experimental condition in Eqn. (4), the wear rate of polyethylene and the LDPE-TD composite with percentage addition of TD petioles can be calculated. The final linear regression equation for the wear rate of the LDPE-TD 500 indicate that model terms are significant. Hence composites when tested against a pin on disc set up can be expressed as follows:

$$W = 0.018 - 0.0073Sd - 0.0095Tt + 0.010ld + 0.015Rr + 0.011SdTt - 0.0079Sdld \quad (5)$$

Table 2, shows the design layout and response data for the wear study. The regression coefficients associated with the variable i.e., load and the reinforcer is positive, which indicate that wear rate increases with increasing load. The TD petioles particles also show significant effect on the wear rate of the composite. Examination of the calculated values of Fishers (F) for all control factors also shows a very high influence of TD petioles additions and applied load on wear rate of the LDPE-TD composite. From Table 3, it can be observed that the additions of TD petioles have the most significant main effect on the wear behaviour of the composites. The interactions effect of sliding distance-time also shows significant effect on the LDPE-TD composites. The model F-value of 2.32 implies the model is significant. There is only a 46.43 % odds that a "Model F-Value" this large could occur due to noise. Value of "Prob F" less than 0.0

this model was used to navigate the design space.

TABLE 2 DESIGN LAYOUT AND RESPONSE DATA FOR WEAR STUDY

Standard order	Sliding distance (m)	Time(sec)	Load(N)	Wear rate at 0%TD	Wear rate at 8%TD
1	70	60	70.68	0.0066	0.0957
2	70	60	70.68	0.0009	0.0051
3	70	120	70.68	0.0068	0.0089
4	70	120	70.68	0.0020	0.0057
5	140	60	141.37	0.0068	0.0154
6	140	60	141.37	0.0041	0.0163
7	140	120	141.37	0.0024	0.0210
8	140	120	141.37	0.0017	0.0227

TABLE 3 ANALYSIS OF VARIANCE TABLE TO IDENTIFY SIGNIFICANT FACTORS INFLUENCING WEAR RATE

Source	Sum of Squares	DF	Mean Square	F Value	Prob>F
Model	0.005146	6	0.0008576	2.32	0.4643
Sd	0.0004292	1	0.0004292	1.16	0.4763
Tt	0.0007182	1	0.0007182	1.94	0.3963
ld	0.0008242	1	0.0008242	2.23	0.3758
Rr	0.001729	1	0.0001729	4.67	0.2758
SdTt	0.0009461	1	0.0009461	2.56	0.3557
Sdld	0.0004993	1	0.0004993	1.35	0.4525
Residual	0.0003699	1	0.0003699		
Cor Total	0.005516	7			

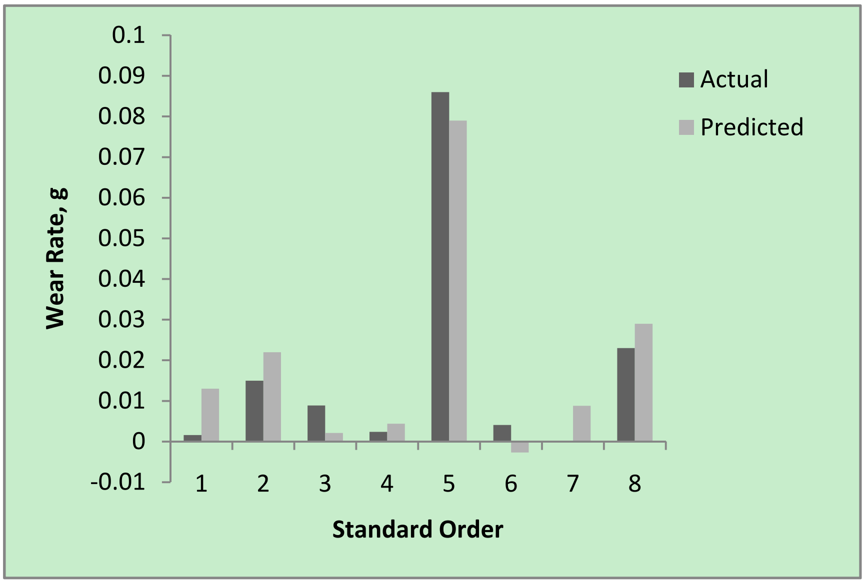

FIG. 10 COMPARISON OF ACTUAL AND PREDICTED MODEL VALUES RESULT

In order to validate the regression model, confirmation wear tests were conducted with parameter levels that were same from those used for the analysis. The different parameter levels chosen for the confirmation tests are shown in Figure 10. The results of the confirmation test were obtained and a comparison was made between the actual wear rate values and the predicted values obtained from the regression model (Figure 10). The error associated with the relationship between the actual values and the predicted values from the regression model for developed composites was very low (less than 3% error). Hence, the regression model developed a demonstrated, feasible and effective way to predict the wear rate of the developed composites.

Conclusions

Polymer matrix composites (PMCs) reinforced with different volume fractions of TD petioles particles were fabricated by means of compressive moulding techniques. The microstructures and wear properties of the composites were characterized and the following conclusions are drawn:

1. Incremental additions of TD petioles increase the mechanical properties such as strength, hardness and modulus with a decrease in impact energy of the LDPE-TD composite.
2. The rate of water absorption rises with increased filler additions. The properties of the composite can be optimized by the additions of anti-clustering agent for the improvement of microstructural homogeneity.
3. The main effect of TD petioles additions and load variable had a pronounced effect of the wear behaviour of the LDPE-TD composite.
4. TD petioles can be used as an agro based filler for the development of LDPE-TD composite

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